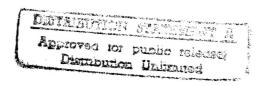
## NASA TECHNICAL NOTE



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PRELIMINARY INVESTIGATION OF FILAMENT-WOUND GLASS-REINFORCED PLASTICS AND LINERS FOR CRYOGENIC PRESSURE VESSELS

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> **DEPARTMENT OF DEFENSE** PLASTICS TECHNICAL EVALUATION CENTER PICATINNY ARSENAL, DOVER, N. J.

#### PRELIMINARY INVESTIGATION OF FILAMENT-WOUND

#### GLASS-REINFORCED PLASTICS AND LINERS

#### FOR CRYOGENIC PRESSURE VESSELS

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#### SUMMARY

A preliminary investigation of the strength characteristics of filament-wound glass-reinforced plastic (FRP) materials was made at temperatures of 75°,  $-320^{\circ}$ , and  $-423^{\circ}$  F. The material was tested in the form of bidirectionally wound sheet material, unidirectionally wound Naval Ordinance Laboratory (NOL) type rings and bidirectionally wound cylinders. Also, a preliminary investigation of metallic and polymeric liners was made in FRP pressure vessels under cyclic strain of  $2\frac{1}{2}$  percent at a temperature of  $-320^{\circ}$  F.

The ultimate strength of FRP materials at -320° and -423° F was significantly higher than that at room temperature. For example, strength increases ranging from 21 to 61 percent were noted for the cryogenic temperatures depending on the material and type of test specimen. The materials were found to be relatively insensitive to sharp notches.

A preliminary investigation of liner materials and liner configurations indicated that aluminum foil liners having some type of provision for mechanical elastic extensibility show promise for use as permeability barriers in FRP pressure vessels subjected to cyclic pressurization resulting in strains of  $2\frac{1}{2}$  percent. In general, plain metallic liners or plain polymeric liners were found to be unsatisfactory.

#### INTRODUCTION

Filament-wound glass-reinforced plastic (FRP) materials offer the highest strength-to-density ratios at room temperature of all the currently available production-type engineering materials. These reinforced plastic materials show strength-to-density advantages that range from about 30 to 100 percent greater than the better aluminum, titanium, or stainless-steel alloys (see ref. 1). Filament-wound reinforced plastic materials have been used successfully for solid propellant rocket motor cases; two of the most outstanding examples are the Polaris and Minuteman missiles (refs. 2 and 3).

Use of filament-wound reinforced plastics would be desirable for liquidoxygen or liquid-hydrogen propellant tanks for space vehicles. In addition to the high strength-to-density characteristics, the reinforced plastics show significant gains in strength at the cryogenic temperatures relative to the strength values at room temperature (ref. 1). These materials also have shown an insensitivity to sharp notches, even at temperatures as low as -4230 F (ref. 1). One problem area that can be anticipated when employing FRP materials to liquid propellant tanks is that related to the porosity of the filament-wound structure. In order for filament-wound pressure vessels to contain liquids or gases, some type of impermeable liner must be employed within the vessel to avoid undesirable leakage of the contained fluid through the filament-wound material. For conventional room temperature applications, an elastomeric material such as natural rubber, silicone rubber, or neoprene is used as an internal permeability barrier. Because these materials are extremely elastic at room temperature, they perform well even when the pressure vessel is highly stressed and elongation or growth of the pressure vessel may be of the order of 2 or 3 percent in length and circumference. The elastomers used for room temperature applications, however, are extremely brittle and inelastic at liquid cryogen temperatures and cannot be used satisfactorily as liners for containing liquid oxygen or liquid hydrogen. Other approaches to the liner problem are therefore required if filament-wound pressure vessels are to be used for storing cryogens. Although no satisfactory solutions have been found for the cryogenic liner problem, reference 4 presents a preliminary study of liner concepts for cryogenic pressure vessels.

Because of the attractive strength characteristics of filament-wound glass-reinforced plastics, the Lewis Research Center has initiated a program for investigating these materials for possible space vehicle applications. Because of the high specific impulses that result from the combustion of hydrogen and oxygen, Lewis is particularly interested in employing these propellants in the propulsion systems of space vehicles. The storage of hydrogen or oxygen aboard space vehicles would be in the liquid state; as a result, there is a need to know the physical behavior of structural materials for propellant tanks at temperatures ranging from about -297° F (boiling point of liquid oxygen at 1 atm) to -423° F (boiling point of liquid hydrogen), as well as room temperature.

The purpose of this report is to present some of the initial results obtained from a long range program for the evaluation of filament-wound glass-reinforced plastics for pressure vessels containing cryogens. Because there is a relatively small amount of data currently available on the behavior of these materials at cryogenic temperatures, it was thought that an interim report would be useful. This report presents initial results obtained on the strength characteristics of filament-wound glass-reinforced plastics for temperatures ranging from about 75° to -423° F. The problem of fluid permeation through the relatively porous walls of the reinforced plastic material is discussed. Some experimental results obtained from investigating the use of polymeric films or metallic foil liners as permeability barriers for the reinforced plastic materials is presented.

The strength characteristics of the materials were investigated at  $75^{\circ}$ ,  $-320^{\circ}$ , and  $-423^{\circ}$  F; flat sheet-type uniaxial test specimens, NOL ring-type specimens, and biaxially stressed model pressure vessels were employed. The

metallic and polymeric permeability barriers (liners) were investigated cyclically at a temperature of  $-320^{\circ}$  F in a biaxially stressed (approximately 1-to-1 circumferential-to-longitudinal stress ratio) model pressure vessel at strains up to about  $2\frac{1}{2}$  percent.

#### MATERIALS

#### Filament-Wound Glass-Reinforced Plastics

The FRP materials considered herein are listed in table I. Both E- and

TABLE I. - FILAMENT-WOUND GLASS-REINFORCED PLASTIC MATERIALS INVESTIGATED

| Configuration                        | Filament<br>disposition | Glass<br>roving<br>(20 end) | Resin                | Hardner                      | Resin<br>content<br>by<br>weight,<br>percent | Composite density, lb/cu in. |
|--------------------------------------|-------------------------|-----------------------------|----------------------|------------------------------|--|------------------------------|
| Bidirectional<br>sheet               | 1 to 1 at 90°           | E-801 <sup>a</sup>          | DER332 <sup>b</sup>  | Methyl<br>Nadic<br>Anhydride | 23.3   | 0.070                        |
| Bidirectionally<br>wound<br>cylinder | 1.8 to 1 at 90°         | E-HTS <sup>a</sup>          | ERL2256 <sup>C</sup> | ZZL0820°                     | 17.5   | 0.076                        |
| NOL ring                             | Uniaxial                | E-HTS                       | EF787 <sup>d</sup>   |                              | 13.9   | 0.077                        |
|                                      |                         | s-hts                       | 660FW <sup>e</sup>   |                              | 16.3   | 0.075                        |

aBy Owens-Corning Fiberglas Corp.

S-types of glass in the form of continuous filaments were considered. As indicated in table I, 801 (an amino silane - see ref. 5) and HTS (an epoxy resin and amino silane - see ref. 5) sizings were employed on the filaments. The filaments were wound into structural composites that were in one of the following forms: (1) bidirectional flat sheets with filaments that were cross wound at a 90° angle with an equal number of filaments in each major direction, (2) NOL ring-type specimens (ref. 6) having only circumferentially wound unidirectional filaments, and (3) bidirectionally wound cylinders having circumferential and longitudinal filaments (in a disposition of 1.8 to 1) that were essentially at a 90° angle. The composite structural materials were obtained from several different commercial fabricators; all fabricators used epoxy resins as the binder material for the glass filaments.

b\_Dow Epoxy Resin by The Dow Chemical Co.

<sup>&</sup>lt;sup>c</sup>By Union Carbide Plastics Co., A Div. of Union Carbide Corp.

dPreimpregnated glass roving by U. S. Polymeric Chemicals, Inc.

ePreimpregnated glass roving by Stratoglas Div., Air Logistics Corp.

#### Liner Materials

The liner materials investigated for use as permeability barriers in the FRP pressure vessels were metallics and polymerics. The metals employed were aluminum and stainless steel in foil form. The polymeric materials investigated were commercially available grades of Mylar A, H-film and Teflon FEP in film

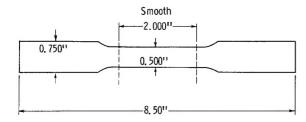
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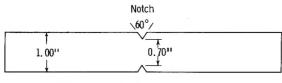
#### APPARATUS AND PROCEDURE

#### Tensile Tests

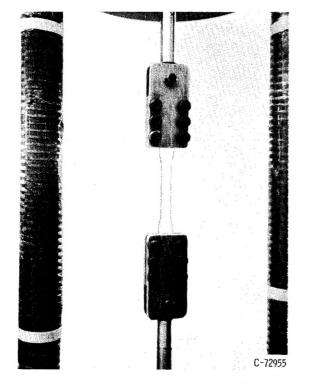
Uniaxial tests of FRP plastic sheets. - Tensile tests of 1/8-inch-thick sheet material were conducted in a universal testing machine with the types of specimen and of grips shown in figure 1. The smooth and notch specimens are shown in figure 1(a). The specimens were cut from the sheet stock so that the longitudinal axis of the specimen was parallel to one of the major directions of the filaments within the sheet material. In the case of the machined notch specimen, the notch angle was 60° and the notch radius was less than 0.001 inch. Figure 1(b) shows a typical smooth specimen mounted in clamp-type grips and ready for a room temperature test. For tests at temperatures of -3200 and -423° F, the specimen was surrounded by a suitable cryostat containing liquid nitrogen or liquid hydrogen. The specimens and grips were in direct contact with the cryogen. A clamp-type extensometer incorporating a linear variable differential transformer was used to measure strain at all test temperatures. The strain rate for all tests was between 0.01 and 0.03 inch per inch per minute.

NOL ring-type tensile tests. - Tensile tests of FRP materials that were in the form of NOL type rings were conducted in a universal testing machine employing the arrangement and grips shown in figure 2. NOL ring-type specimens with an inside diameter of 5.75 inches as shown in figure 2(a) were inserted into split-disk-type grips as shown in figure 2(b). The split disk was cut from an original one piece disk that had an outside diameter of 5.75 inches. When the ring specimen was inserted into the split-disk fixture, the gap between the two disk halves was about 1/16-inch before a load was applied to the assembly. With this arrangement, the rings were subjected to an essentially uniaxial tensile loading across the small gap between the two disk halves. Figure 2(b) shows an NOL ring specimen mounted in a split-disk fixture in which the tensile load is transmitted through a pin in each disk half. The test setup shown in figure 2(b) is for use at room temperature; for cryogenic temperatures, the specimen and grip assembly was submerged in a cryogen contained within a cryostat. In order to promote uniform slippage of the ring inner surface with respect to the ring loading surface (due to strain within the specimen), the ring loading surface of the split-disk fixture was lubricated with petroleum jelly for tests at room temperature and with Teflon TFE powder at cryogenic temperatures. Universal joints (for alinement purposes) were included in the tensile loading system for all tests. The strain rate was between 0.01 to 0.03 inch per inch per minute.



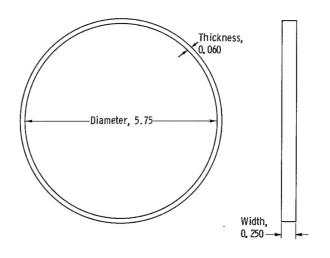


(a) Smooth and notch sheet specimens.

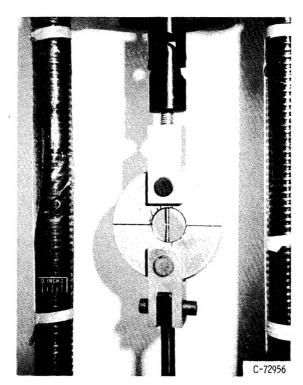


(b) Sheet specimen mounted in tensile machine.

Figure 1. – Tensile test of 1/8-inch-thick bidirectionally wound flat sheet.

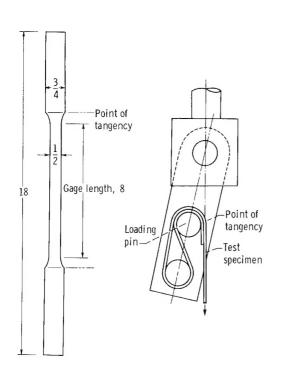


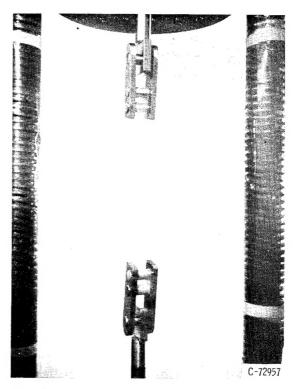
(a) NOL ring specimen.



(b) NOL ring mounted for tensile test.

Figure 2. - NOL ring tensile test. (All dimensions in inches.)





(a) Polymeric film test specimen.

(b) Self-locking film grips.

(c) Typical specimen mounted in grips for room temperature test (specimen placed in cryostat for -320° and -423° F tests).

Figure 3. - Tensile specimen and fixtures used in uniaxial tests of polymeric film materials. (All dimensions in inches,)

Strain in the NOL rings was determined by the gap growth between the two disk halves. A linear variable differential transformer was used to measure gap growth at all test temperatures. The total strain was taken as two times the gap growth, and the total strain was assumed to be distributed uniformly throughout the mean circumference of the ring.

Uniaxial test of polymeric films. - The tensile strength of the polymeric film materials was evaluated in a universal testing machine using the test arrangement shown in figure 3. Strips of the film material that were initially about 18 inches long and had a test section of 8 inches as shown in figure 3(a) were inserted into self-locking grips as shown in figure 3(b). The film specimens were inserted into the self-locking grips so that the portion of the specimen where the decrease in width started was tangent to the loading pin (see fig. 3). A typical setup for a test at room temperature is shown in figure 3(c). For temperatures of -320° and -423° F, the specimens and gripping fixtures were submerged in liquid nitrogen or liquid hydrogen contained within a cryostat as mentioned previously. Strain for all temperature conditions was determined by measurement of the movement of the tensile machine head. Strain rates were about 0.05 inch per inch per minute.

#### Cylinder Burst Tests

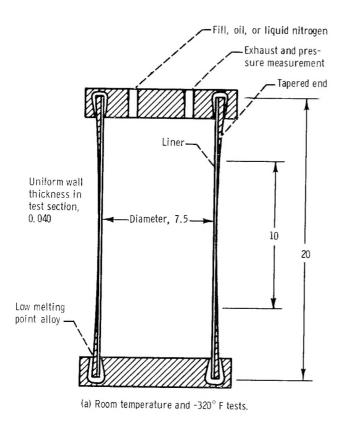
The bidirectional test specimens selected for investigating the burst

strength of FRP pressure vessels were cylinders of the type shown in figure 4. The cylinders had an inside diameter of 7.5 inches, a length of 20 inches, and a nominal test section wall thickness of 0.040 inch. Because of the inherent porosity of the FRP when strained, 5-mil 1100-0 aluminum liners were installed to allow pressurization to burst. Aluminum was chosen as the liner material since it has high elongation at cryogenic temperature (55 percent at -320° F according to ref. 7). Since the liners were made from foil sheets, it was necessary to have a seam in the liner. A material overlap of 1/2 inch was chosen. The seams were sealed with an epoxy adhesive (see table II) after the liner was fitted into the FRP tube. A rubber bladder was inserted into the test vessel and pressurized to support the liner and keep the seam region under pressure during the adhesive curing process. Generally, the liners were not attached to the inside surface of the pressure vessel so they were essentially free floating in the test section.

Inasmuch as the test cylinders had open ends, a type of end closure such as that shown in figure 4 had to be provided. These end closures were removable and reusable and were similar in concept to those described in references 8 and 9 for metal pressure vessel investigations. The end sections of the reinforced plastic tubes were tapered as indicated in figure 4; a portion of the tapered end section fit into a circular groove in each end cap. In assembly, the clearance volume between the test specimen and circular grooves in the end caps was filled with a molten low melting point alloy; upon solidifying, the alloy expanded and effectively locked and sealed the end caps securely to the test specimen.

Two different low melting point alloys were used, namely, Cerromatrix (melting range of 218° to 440° F) and Cerrobend (melting point of 158° F). (Cerromatrix and Cerrobend are manufactured by Cerro Corp.) Cerromatrix was used for test cylinders that were to be investigated at 75° F because the strength characteristics of this alloy were higher than those of Cerrobend at room temperature conditions. Cerrobend was used for test cylinders that were to be investigated at -320° and -423° F where the strength characteristics of of this alloy were sufficient. Also, the lower melting point of Cerrobend was thought to be advantageous in that it did not result in the liner materials (particularly, the polymeric films) or the seam adhesives being exposed to unusually high temperatures during the assembly process. Figure 5(a) shows an exploded view of a typical test vessel.

Hoop strain was measured by the deflection of a strain-gaged cantilever beam that was actuated by a 10-mil steel wire circumscribing the test cylinder at the midpoint of the test section. One end of the wire was rigidly fixed relative to the cylinder, while the other end was secured to the end of the cantilever beam. The installation is shown in figure 5(b). Hoop growth of the cylinder resulted in a tensile load in the wire, which in turn caused deflection of the beam. A calibration relating tip deflection of the beam to the strain-gage reading determined the hoop growth. The strain rate varied to some degree depending upon the temperature level at which the tests were being made. For the room temperature tests, the strain rate was about 0.01 inch per inch per minute; while for temperatures of -320° and -423° F, the strain rate was about 0.005 inch per inch per minute. The slower loading rate for the cryogenic temperature levels was dictated by the operating characteristics of the cryogenic



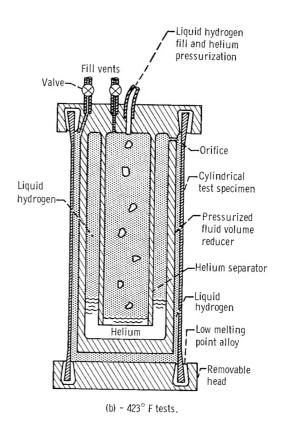


Figure 4. - Schematic diagram of biaxial cylindrical filament-wound glass-reinforced plastic test specimens with removable and reusable end caps used for room temperature, -320°, and -423° F tests. (All dimensions in inches; not to scale.)

TABLE II. - SUMMARY OF TESTS ON METALLIC LINERS AT -320° F

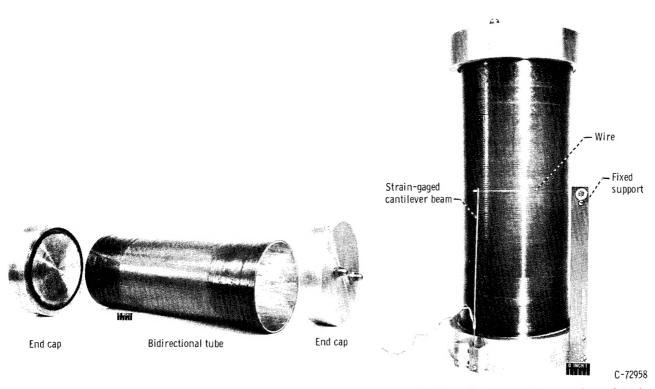
| Material                                   | Type of pattern | Thickness, | Adhesive                  | Backup<br>material | Cycles to failure at $2\frac{1}{2}$ -percent strain in test cylinder |
|--|-----------------|------------|---------------------------|--------------------|--|
| AISI 347 Stain-<br>less steel <sup>a</sup> | Plain           | 0.001      | Polyurethane <sup>b</sup> |                    | 8  |
| 1100-0 Aluminum <sup>C</sup>               | Plain           | .005       | Epoxy <sup>d</sup>        | ~                  | 1  |
|  | Spiral          |            |                           | Teflon felt        | 5  |
|  | Waffle          |            |                           | Dacron felt        | 1  |
|  | Zigzag          |            |                           | Dacron felt        | 5  |
|  | Sinusoidal      | 1          | 1                         | 0.020-inch wire    | 18   |

<sup>&</sup>lt;sup>a</sup>Liner attached to tube with adhesive.

 $<sup>^{\</sup>rm b}\text{Adiprene-Ll00}$  and MOCA by E. I. du Pont de Nemours & Co.

<sup>&</sup>lt;sup>c</sup>Liner unattached.

 $<sup>^{\</sup>mbox{\scriptsize d}}\mbox{\it Epon}$  815 and Curing Agent T-1 by Shell Chemical Corp.



(a) Exploded view of major components.

(b) Assembled test cylinder with strain-measuring equipment.

Figure 5. - Typical bidirectional test cylinder.

test apparatus. A low viscosity oil or water was used as the pressurizing liquid for the room temperature tests. For the -320° and -423° F tests, liquid nitrogen and liquid hydrogen, respectively, were used as the pressurizing liquids and the test vessel was installed in a cryostat to reduce the liquid cryogen boiloff rate. The burst test at cryogenic temperatures were made in the same manner as described in detail in reference 8.

#### Cyclic Tests of Liners

The investigation of the cyclic biaxial strain characteristics of various liner materials at -320° F was conducted in a FRP cylinder similar to, but of different construction, than those discussed in the previous section. The cylinders for liner studies were made of inexpensive commercially available helically wound (filaments oriented at an angle of 54.7° to the longitudinal axis) tubing with glass-cloth reinforcements to provide the tapered ends (see fig. 4). Liners of various materials and structural configurations were then placed inside the tube and caps were attached to the ends of the tube in the same manner as that described in the section Cylinder Burst Tests. The completed test specimen was the same as that shown schematically in figure 4.

Inasmuch as glass-reinforced plastic materials are capable of strain values greater than 3 percent before rupturing, working biaxial strains of the order of  $2\frac{1}{2}$  percent were chosen for the cyclic strain investigation of liners

TABLE III. - SUMMARY OF TESTS ON LAP SEAM

POLYMERIC LINERS AT -320° F

| r                   | 9 7                     |
|---------------------|-------------------------|
| Adhesive            | , epoxy <sup>a</sup> .] |
| [ - accric to the c | 9 CPC25, 9 1            |

| Material  | Thickness,                                    | Maximum curing temperature,  OF     | Maximum<br>strain at<br>failure,<br>percent<br>(b) |
|---|---|-------------------------------------|--|
| Mylar A <sup>c</sup> ,d<br>Mylar A <sup>e</sup><br>Mylar A <sup>e</sup><br>Mylar A <sup>e</sup><br>H-film <sup>c</sup> ,d<br>H-film <sup>e</sup><br>H-film <sup>e</sup> | 0.002<br>.003<br>.002<br>.002<br>.003<br>.003 | 150<br>150<br>150<br>75<br>75<br>75 | 1.0<br>1.1<br>1.2<br>1.5<br>1.0<br>1.6<br>1.5      |

<sup>&</sup>lt;sup>a</sup>Epon 815 and Curing Agent T-1 by Shell Chemical Corp.

investigated herein. In order to achieve such strain values for the 0.040-inch-thick reinforced plastic test specimen, liquid pressures of the order of 800 to 1000 pounds per square inch were required. It was realized that such high working pressures are unrealistic for actual reinforced plastic cryogenic propellant tanks for space vehicle application. Furthermore such pressures might result in misleading test results for certain liner materials and liner concepts. As a result, the use of the relatively thickwalled (0.040-inch) FRP test specimen was considered suitable for liner screening purposes only.

Polymeric liners. - The polymeric liner materials considered were Mylar A, H-film, and Teflon FEP. Each of the materials was obtained in a sheet film form and then fabricated into tensile test specimens or liners. The sheet thickness of these materials ranged

from about 0.001 to 0.003 inch. These polymeric liner materials were cut to the proper size and then fitted to the inside diameter of the test cylinder. The polymeric liner materials were formed and installed with a lapped seam. A material overlap of 1/2 inch was used; the seam was sealed with an epoxy adhesive (see table III).

The test procedure was to first fill the test vessel with the liquid nitrogen and then to slowly increase the pressure within the vessel until a strain of about  $2\frac{1}{2}$  percent was achieved in the cylinder wall. The pressure was then slowly decreased to ambient conditions and the loading-unloading cycle repeated. This cyclic pressure procedure was repeated until the liner failed. The strain rate was about 0.005 inch per inch per minute.

Metallic liners. - Aluminum foil (type 1100-0) having a nominal thickness of about 0.005 inch was used for all of the investigations of metallic liners except one; in this instance, stainless-steel foil was used. Initially, plain flat aluminum foil was employed, but as the investigation progressed, aluminum foils having various types of embossed patterns were also investigated. These embossed patterns will be discussed more fully in the section RESULTS AND DISCUSSION.

The procedure for installing a given metallic foil liner in a test cylinder was similar to that for the burst tests discussed in the section Cylinder Burst Tests. The aluminum liners were free floating, and had a single longitudinal seam that was bonded by an epoxy adhesive.

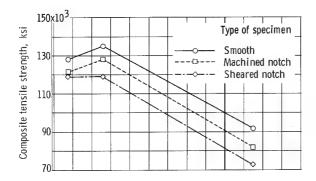
The test procedure for the metallic liners was the same as that mentioned

bFailed on initial cycle.

 $<sup>^{\</sup>text{C}}\text{Manufactured}$  by E. I. du Pont de Nemours & Co.

 $<sup>^{</sup>m d}$ Liner attached to tube with adhesive.

<sup>&</sup>lt;sup>e</sup>Liner unattached.



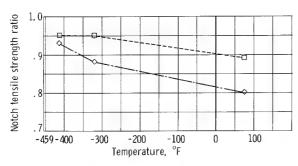


Figure 6. - Strength characteristics as function of temperature for bidirectionally wound glass-reinforced plastic sheet material (E-801 glass).

in the previous section for the polymeric liners.

#### RESULTS AND DISCUSSION

Strength Characteristics of Filament-Wound

Reinforced Plastic Materials

Bidirectional glass-reinforced sheet material. - A summary of the smooth and notch tensile strength data obtained at temperatures of 75°, -320°, and -423° F for the bidirectional glass-reinforced plastic sheet material is given in table IV and plotted in figure 6. The strength was calculated based upon the total cross-sectional area of the specimen in the smooth or notched region. It can be seen from figure 6 that there is considerable increase in actual strength of the material as the temperature is decreased from 75° to -423° F. This is true for each of the three different types of

TABLE IV. - SUMMARY OF NOTCH TENSILE STRENGTH OF BIDIRECTIONALLY WOUND REINFORCED PLASTIC SHEET MATERIAL (E-801 GLASS FIBER)

| Type             | Test temperature, <sup>O</sup> F                         |  |  |      |                              |      |  |
|------------------|--|--|--|------|------------------------------|------|--|
| of<br>specimen   | 75   | -320   | -423                                     | 75   | -320                         | -423 |  |
|                  | Composite  | Composite tensile strength, psi                                |  |      | Notch tensile strength ratio |      |  |
| Smooth           | 87,700<br>97,500<br>93,700<br>90,200<br>87,600<br>92,000 | 140,000<br>137,200<br>132,000<br>128,700<br>139,000<br>133,000 | 126,200<br>128,200<br>131,000<br>127,000 |      |                              |      |  |
| Average          | 91,500   | 135,000  | 128,000                                  |      |                              |      |  |
| Machined notch   | 84,000<br>83,000<br>77,900                               | 124,000<br>125,000<br>134,000                                  | 120,000<br>121,000<br>121,000            |      |                              |      |  |
| Average          | 81,600   | 128,000  | 121,000                                  | 0.89 | 0.95                         | 0.95 |  |
| Sheared<br>notch | 73,600<br>72,000<br>72,700                               | 122,000<br>121,000<br>115,000                                  | 123,000<br>110,000<br>125,000            |      |                              |      |  |
| Average          | 72,800   | 119,000  | 119,000                                  | 0.80 | 0.88                         | 0.93 |  |

specimens represented. The smooth specimen strength increases from 91,500 psi at  $75^{\circ}$  F to 128,000 psi at  $-423^{\circ}$  F, an increase of about 40 percent. At  $-320^{\circ}$  F, the smooth tensile strength was 135,000 psi, an increase of about 47 percent above that at  $75^{\circ}$  F.

Of equal importance is the behavior of the material in a notched condition, which is also indicated in figure 6. It can be seen that the machined and sheared sharp notch specimens exhibited strengths that were lower than those of the smooth specimens but that the notch strength essentially paralleled that of the smooth ones. For the machined notch specimens, the strength was about 7000 to 10,000 psi less than strengths for the smooth specimens throughout the temperature range. The sheared notch specimens showed strengths that were about 9000 to 19,000 psi less than those of the smooth specimens throughout the temperature range.

The ratio of the sharp notch to smooth tensile strengths is plotted in figure 6. It can be seen that the machined notch specimens had a ratio of notch to smooth tensile strengths that ranged from about 0.89 at 75° F to 0.95 at -320° and -423° F. For the sheared sharp notch, the values of this ratio were somewhat lower ranging from 0.80 to 0.93. The important fact to be observed from the notch specimen data is that the reinforced plastic material did not exhibit a significant notch sensitivity at any temperature level. Actually, there was a tendency for the notch sensitivity to decrease with decreasing temperature, which is in marked contrast to the behavior of most metal alloys. Alloys generally show an increase in notch sensitivity as the temperature is decreased; for example, reference 1 shows that certain alloys of aluminum and titanium may have notch strengths that are only 30 to 60 percent of their smooth tensile yield strength at -423° F.

The reason for the sheared sharp-notch specimens fracturing at a lower strength than the machined sharp-notch specimens is not clearly understood. It may be caused by the fact that the sheared specimens do not exhibit the clean cut that the machined notch specimens do. In the shearing process, a region adjacent to the notch was damaged, and this may effectively reduce the material strength in this region. This type of shear specimen was used because it was felt that it might more nearly approach the type of ragged edge failure that would result if a reinforced plastic structure were penetrated by a blunt tool or similar object.

The very good notch characteristics exhibited by the sheet-type reinforced plastic specimens, particularly those at cryogenic temperatures, may be reasonable to expect when the composite structure is considered. Glass exhibits a brittle characteristic at room temperature that probably cannot be expected to change markedly with further decreases in temperature. Also, in a composite structure of glass fibers and plastic, a crack would probably not be as likely to propagate as in a homogeneous material. The reason for this is that in a glass fiber composite a given crack or flaw would be expected to propagate only across an individual fiber (having a diameter of the order of 0.0004 inch) leaving this particular fiber ruptured and unable to carry any further load in the vicinity of the fracture, but in turn transferring its share of the load to the balance of the fibers in the structure without further propagation of the crack.

TABLE V. - SUMMARY OF TENSILE STRENGTH OF FILAMENT-WOUND GLASS-REINFORCED NOL RINGS AND BIDIRECTIONALLY WOUND CYLINDERS

| Configuration   | Glass roving | Test temperature, oF  |                               |  |  |
|---|--------------|---|-------------------------------|--|--|
| Conriguration   | (20 end)     | Test temperature, F   |                               |  |  |
|   | (=0 0.1.4)   | 75  | -320                          | -423   |  |
|   |              | Tensil  | e streng                      | th, psi  |  |
| NOL ring  | S-HTS        | 301,000<br>297,000<br>274,000<br>306,000<br>304,000<br>298,000<br>314,000<br>307,000<br>276,000 | 372,000<br>374,000<br>371,000 | 366,000<br>391,000<br>346,000<br>358,000<br>341,000<br>357,000 |  |
|   | Average      | 297,000   | 373,000                       | 360,000  |  |
|   | E-HTS        | 214,000<br>198,000<br>233,000   |                               |  |  |
|   | Average      | 215,000   | 295,000                       |  |  |
| Bidirectionally wound cylinder (hoop composite)       | E-HTS        | 194,000   | 301,000<br>301,000<br>300,000 | 293,000  |  |
|   | Average      | 197,000   | 301,000                       | 293,000  |  |
| Bidirectionally wound cylinder (total wall thickness) | E-HTS        | 124,000   | 205,000<br>205,000<br>204,000 | 198,000  |  |
|   | Average      | 127,000   | 205,000                       | 198,000  |  |

NOL ring and cylinder burst tests. - Tensile properties of FRP materials were also determined by using NOL ring-type specimens and bidirectional cylinders as described previously in the section APPARATUS AND PROCEDURE. The results of individual tests are tabulated in table V. Figure 7 shows the average composite tensile strengths of the various test configurations and materials as a function of temperature. The superiority of the tensile strength of S-HTS glass relative to that of E-HTS glass is evident at all test temperatures. The composite tensile strengths of NOL rings of S-HTS glass were 297,000, 373,000, and 360,000 psi at 75°, -320°, and -423° F, respectively. This is equivalent to about a 26- and 21-percent increase in strength at -320° and -423° F, respectively, relative to that at 75° F. For NOL rings of E-HTS glass the tensile strength was 215,000 and 295,000 psi at 75° and -320° F, respectively.

Bidirectional FRP cylinders of E-HTS glass show strength trends similar to that of the NOL ring tests. Based on only the composite hoop cross section of the cylinders, the strength agrees well with that of the NOL rings down to  $-320^{\circ}$  F. Based on the total wall thickness, the tensile burst strengths were

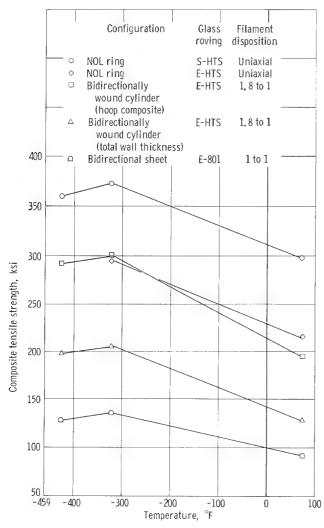


Figure 7. - Composite tensile strength of filament-wound glassreinforced plastic NOL rings and bidirectionally wound cylinders and sheet as function of temperature.

127,000, 205,000 and 198,000 psi at  $75^{\circ}$ ,  $-320^{\circ}$ , and  $-423^{\circ}$  F, respectively. This is equivalent to a 61- and a 56-percent increase in strength at  $-320^{\circ}$  and  $-423^{\circ}$  F, respectively, relative to that at  $75^{\circ}$  F.

Although no bidirectional cylinders of S-HTS glass were tested in this investigation, a strength increase of about 25 percent over that of E-HTS cylinders would be predicted based upon the E-HTS NOL ring tests.

The lower curve of figure 7 is repeated from the smooth specimen data for FRP sheet material of figure 6; it is presented again in figure 8 for the convenience of making strength comparisons. Because the E-801 glass was wound into a bidirectional sheet having filaments at right angles to each other in a 1-to-1 ratio, the sheet strength would be doubled if only the composite thickness in the direction of loading were considered. For example, at 750 and -320° F, the bidirectional sheet in smooth specimen form (see fig. 6 or bottom curve in fig. 7) had a strength of 91,500 and 135,000 psi, respectively. If these values were doubled (because of the 1-to-1 filament ratio), the strength at 750 and  $-320^{\circ}$  F would be 193,000 and 270,000 psi, respectively. These would com-

pare to values of about 200,000 and 300,000 psi at temperatures of 75° and -320° F, respectively, for the E-HTS glass in the form of NOL rings or the hoop composite of bidirectionally wound cylinders. Thus, the E-HTS glass is predicted to be about 7000 psi stronger than the E-801 glass at 75° F. At -320° F, the E-HTS glass is predicted to be about 30,000 psi stronger than the E-801 glass. From these predictions it appears that there is a definite advantage in strength for the E-HTS glass with respect to E-801 glass at -320° F.

It should be noted that the various test configurations and materials show a wide latitude in strength. Variables such as glass composition and sizing would be expected to affect strength. However, other variables that may have contributed to the higher strength of materials in NOL ring or bidirectional cylinder form may be the method of fabrication (a circular wound specimen against a wound flat plate), the type of epoxy resin employed, the winding tensions employed, the glass-resin content, plus the various techniques that

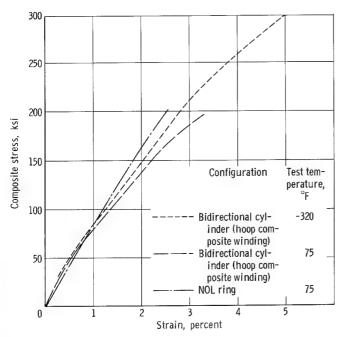


Figure 8. – Stress-strain diagrams of E-HTS glass in form of NOL rings and bidirectional cylinders at  $75^\circ$  and –320° F.

different fabricators undoubtedly employ in the fabrication process. It is beyond the scope of this report to investigate the effects these variables may have upon the strength of the material, but it should be realized that reinforced plastics, like metal alloys, can be fabricated and processed in many ways and that the processing variables can strongly influence the mechanical properties of the finished material.

Strain characteristics of FRP. - Stress-strain diagrams are shown for NOL rings and bidirectional cylinders in figure 8. For comparison with the NOL rings, the stress of only the hoop filaments of the bidirectional cylinders is shown. It is seen that the stress-strain relation is not linear,

particularly as the fracture point is approached. This nonlinearity is more pronounced in the cylinders than in the NOL rings. The initial moduli of the unidirectional composites are of the order of 8,000,000 to 8,500,000 psi with only a minor increase in modulus occurring when the temperature is decreased from 75° to -320° F. Of particular interest is the significant increase in fracture strain (up to 5 percent) at cryogenic temperatures.

Cyclic Characteristics of Metallic and Polymeric Liner Materials in

#### Reinforced Plastic Pressure Vessels

General considerations for cyclic liner materials. - As discussed in the previous section on strain characteristics, the unidirectional composite modulus of elasticity of reinforced plastic materials is low, of the order of 8,000,000 to 8,500,000 psi. Material elongations at failure of the order of 3 to 5 percent are obtained for FRP. Thus a liner for a reinforced plastic pressure vessel that is to undergo a single pressure loading to failure of the reinforced plastic wall must have an extensibility of about 3 to 5 percent without failure. Liners for cyclic pressurization must have other characteristics, which will be discussed in greater detail in the following paragraphs.

For practical applications, the elongation at pressure vessel burst is not of prime importance, but the elongation at some reasonable working stress of the structural material is of more significance. If FRP materials were to be used for the tanks of space vehicles, working stresses of the order of 75 percent (ref. 3) of the ultimate strength would probably be reasonable. From data presented previously, such working stresses would result in material elongations

of the order of 2 to  $3\frac{1}{2}$  percent. For any practical tank application, the pressure vessel would have to be cycled a number of times in its useful lifetime. The number of pressure cycles that a given tank might have to undergo would vary with the particular application, the mission of the vehicle in which it was being used, and the type of proof testing that would be required to assure vehicle reliability. It is difficult to specify the exact number of cycles that a cryogenic pressure vessel for space application may have to withstand, but for the purposes of this investigation it was arbitrarily assumed that a FRP pressure vessel and its associated impermeable liner has to withstand a minimum of 25 pressure cycles to be considered satisfactory.

In addition to the elastic strain capabilities, a liner for space vehicle application should be lightweight, capable of being employed successfully in a large range of tank sizes and configurations, and compatible with the processes by which filament-wound pressure vessels are fabricated. In addition, the liner material must be chemically compatible with the cryogens to be contained.

Material considerations. - For this initial investigation on possible liner concepts and materials, aluminum (type 1100-0) was considered as a likely metallic liner candidate. This choice was made partly on the basis of the success obtained in using aluminum foil liners for the single pressurization burst tests described previously. Although aluminum foil does not have the necessary elastic extensibility for repeated strains of 2 to  $3\frac{1}{2}$  percent, the full-soft foil might, because of its high ductility, be suitable in its plain sheet form; or if that were not suitable, a modified embossed form of aluminum foil might prove successful. This concept will be discussed in more detail in the section Metallic liners. In one instance, plain stainless-steel (type AISI 347) foil was also investigated as a possible liner material.

Polymeric films may also offer some prospect of success for liner applications. Certain polymeric films have very low permeability, some are chemically compatible with hydrogen, and some are relatively inert to oxygen. In addition, a polymeric film liner would add very little weight compared to metal alloys. Data on the physical characteristics of polymeric films at cryogenic temperature levels, however, is meager. Three polymeric films were considered for an initial screening investigation. These films were commercially available forms of (1) Mylar A, (2) H-film, and (3) Teflon FEP. They were selected for their availability, their low permeabilities, and their relative ease of fabrication into test liners.

Table VI summarizes the results of a brief screening of the uniaxial mechanical properties of the three film-type materials used for liner applications. These tests were intended only to act as a guide and were not comprehensive; the FEP material because of its high thermal contraction and low strain at -320° F was investigated only at this temperature. The Mylar A and H-film materials appeared to have suitable strain characteristics at -320° F (of the order of 6.5 to 7 percent), while the Teflon FEP material exhibited only a 3.1 percent strain. The Mylar A material showed a total strain of 5.5 percent at -423° F.

# TABLE VI. - SUMMARY OF UNIAXIAL MECHANICAL PROPERTIES OF POLYMERIC FILMS SELECTED FOR INVESTIGATION AS LINER MATERIALS IN FILAMENT-WOUND GLASS-REINFORCED

#### PLASTIC PRESSURE VESSELS

| Polymeric<br>film | Test<br>temper-<br>ature,<br>°F | Tensile<br>strength,<br>psi | Thermal<br>contraction<br>from 75° F<br>to test tem-<br>perature,<br>percent | Modulus of elasticity, psi    | Total<br>strain,<br>percent |  |  |  |
|-------------------|---------------------------------|-----------------------------|--|-------------------------------|-----------------------------|--|--|--|
| Mylar A           | 75<br>-320<br>-423              | 22,000<br>45,000<br>50,000  | 0.36   | 500,000<br>900,000<br>950,000 | 90.0<br>7.1<br>5.5          |  |  |  |
| H-film            | 75<br>-320<br>-423              | 25,000<br>43,000            | 0.45   | 450,000<br>1,000,000          | 70.0<br>6.5                 |  |  |  |
| Teflon FEP        | 75<br>-320<br>-423              | 3,000                       | 1.80   | 45,000<br>600,000             | 300.0<br>3.1                |  |  |  |

The thermal contraction for the three polymeric materials was obtained only for the range of temperatures from 75° to -320° F. The values of this factor for the Mylar A and H-film materials were reasonably low, being 0.36 and 0.45 percent, respectively. The thermal contraction for the Teflon FEP material was 1.80 percent. Unpublished NASA data indicated that the thermal contraction for filament-wound glass-reinforced plastics are of the order of 0.05 percent. Thermal contraction characteristics for liner materials that are similar to those of the FRP structural material are desirable so that excessive dimensional changes between the structure and the liner do not result from temperature reductions caused by filling the tank with a cryogen. Any contraction of the liner material that is greater than the contraction of the structural material will result in amplifying the elastic extensibility problem already present. The relatively high thermal contraction for the Teflon FEP material combined with its relatively low strain capabilities at -320° F made this material appear to be a rather doubtful candidate for successful liner application; it was therefore eliminated for further consideration as a cryogenic liner material. As a result the only polymeric materials fabricated into liners were Mylar A and H-film. The fabrication of these materials into liners and their installation into the test vessels is described in the section APPARATUS AND PRO-CEDURE.

Polymeric liner results. - Four liners of Mylar A and three liners of H-film materials were investigated at a temperature of -320° F using the test apparatus described previously. The Mylar A material was 0.002- and 0.003-inchthick plain film. A longitudinal seam made with epoxy adhesive cured at 150° F was employed in the liner. No backup material was employed between the liner and the filament-wound reinforced plastic structure. The H-film liners were

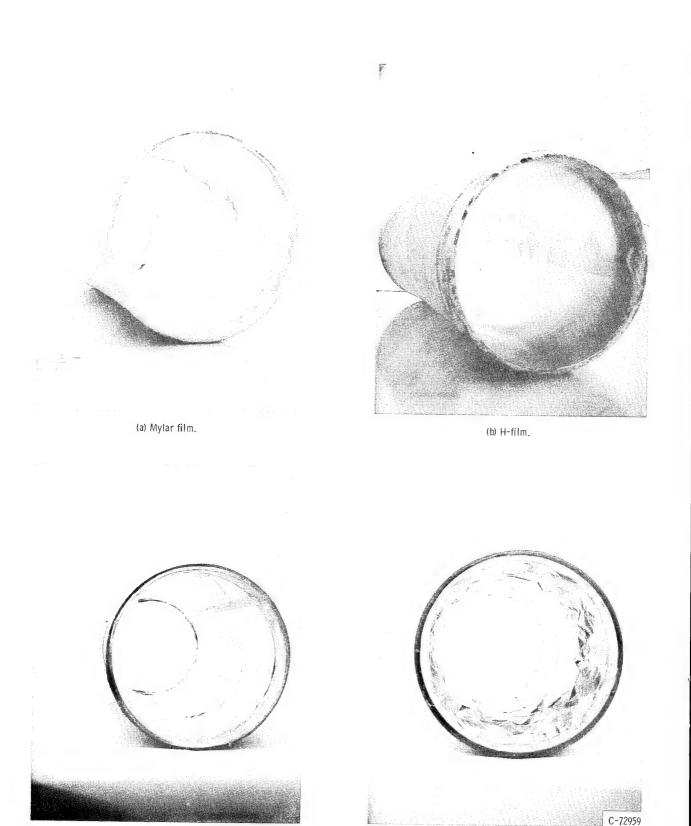


Figure 9. – Photographs of cylindrical, plain surface liners after repeated pressurization at -320  $^{\circ}$  F.

(d) Stainless steel.

(c) Aluminum foil.

0.003-inch thick and made from plain film material. A longitudinal seam sealed with epoxy adhesive cured at  $75^{\circ}$  F was used; no backup material was employed. All liners were free floating with the exception of one Mylar A and one H-film liner that were adhered to the inside surface of the FRP tube. The major construction details and the test results for these liners are summarized in table III (p. 10). Typical failures of Mylar A and H-film liners are shown in figures 9(a) and (b).

It was intended that these liners would be cyclically strained to a value of about  $2\frac{1}{2}$  percent with liquid nitrogen as the pressurizing liquid. All seven liners failed, however, at strains of the order of 1 to  $1\frac{1}{2}$  percent during the first pressurization cycle. There did not appear to be any advantage in using a bonded liner over a free floating liner. Based upon the preliminary tests made of the unidirectional elongation characteristics of the films, failure at strains as low as 1 to 1.6 percent were not expected. Apparently the biaxial straining of the polymeric film-type liner materials in the pressure vessel has some effect on its strain characteristics. In order to further check the possibility that the biaxial straining of the liner materials had an adverse effect on its cyclic durability, a modified test assembly was employed for several tests. External longitudinal tie rods that connected the removable and reusable heads were employed to restrain the test tank ends with the result that, upon pressurization of the test tank, the longitudinal strain in the tank and liner materials was essentially zero. Three Mylar A liners were tested with this arrangement. A cyclic life of 1, 4, and 7 cycles was attained with the Mylar A material strained uniaxially (hoop direction only) to a value of  $2\frac{1}{2}$  percent.

Although the polymeric film materials investigated herein were not suitable for cryogenic liner application, it should be realized that these were commercially available types of materials that were not formulated and processed with the intent that the materials be used at cryogenic temperatures. Materials such as those used herein can be formulated and processed in a number of ways that may influence the cryogenic properties of the material in a significant manner. For example, reference 10 indicates that the degree of crystallinity of Mylar A type of material can greatly influence the extensibility of the material at cryogenic temperatures. The types of films used herein were known to have some degree of orientation applied to them in their commercial fabrication processing; such orientation may have an effect on the bidirectional properties of the materials. Also, the processing temperatures involved in preparing the liners and the pressure vessels (150° F to cure the liner seam adhesives and about 160° to the removable and reusable end closures on the test cylinders) may have a tendency to relieve the orientation or change the crystallinity of the materials. With the wide latitudes available in the formulation and processing of these materials it may be possible to evolve a suitable polymeric film material for cryogenic liner application in conjunction with FRP pressure vessels.

Metallic liner results. - Table II (p. 8) summarizes the results that were obtained by investigating plain liners made from aluminum or stainless steel and from more complex aluminum liners that incorporated embossed concepts in an attempt to provide greater extensibility than was available in the plain types

of liners. For all the liner tests the pressure vessel was pressurized with liquid nitrogen to achieve a strain of  $2\frac{1}{2}$  percent in the tank wall in both the circumferential and longitudinal directions. The results are discussed in greater detail in the following sections.

Plain liners: Although neither aluminum nor stainless steel have elastic strain capabilities of the desired 2 to  $3\frac{1}{2}$  percent for use in reinforced plastic pressure vessels, these materials do have plastic elongations to failure at cryogenic temperatures that are well beyond the 2 or  $3\frac{1}{2}$  percent requirement. It was thought that even though the materials would be strained beyond their yield points during the first pressurization cycle, the buckling that would occur when the pressure was decreased to zero, might not be so serious that the foil liner materials would fail upon subsequent pressure cycling.

From table II it can be seen that a plain aluminum liner made from 1100-0 aluminum that was 0.005-inch thick failed after only 1 cycle; actually the liner failed after pressurization for the second cycle had started. The pressure was relatively low and the strain at which the failure occurred during the second cycle was less than 1 percent. A photograph of the failed liner after removal of the end closures is shown in figure 9(c); it can be seen that the liner buckled randomly and, in some cases, quite severely.

A plain liner made from 0.001-inch-thick type 347 stainless steel failed after 8 cycles. The condition of this liner is shown in figure 9(d); it can be seen that it buckled randomly in a manner that appears to be more severe than that for the aluminum liner. The buckling observed in figure 9(d) is probably more severe than that which actually occurred at failure; the severe buckling results from liquid and gas getting between the liner and the inside surface of the reinforced plastic tube as a result of the liner failure. When the internal pressure is released, the liquid cryogen may flash into a gas and force the liner radially inward, which results in the appearance of buckling that is believed to be more drastic than that which actually existed at the time of failure.

The 8 successful cycles that were obtained with the stainless-steel liner might have been due in part to the buckling of the material. Assuming that the majority of the buckling was imposed upon the liner after the pressure was reduced to zero at the conclusion of the first cycle, the liner probably had many small wave-like buckled regions in the liner. When the liner was pressurized on successive cycles, it may have been possible that the smaller amplitude and smaller frequency waves did not become completely flattened under the influence of the internal pressure. If this were so, the buckled regions may have acted as hinges and permitted the liner to have some elastic extensibility up to the  $2\frac{1}{2}$ -percent strain condition. Because of its higher strength and modulus, it may have been conceivable for the stainless-steel material to resist the flattening action of the buckled regions better than the aluminum material and thus show better performance for the stainless-steel liner than for the aluminum liner. In any event, the stainless-steel liner failed eventually in the same general

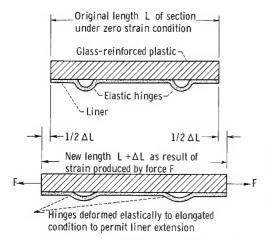


Figure 10. - Schematic diagram of liner design that permits liner to elongate.

manner as the aluminum liner, that is, from excessive straining and buckling in an uncontrolled fashion that results in early severe fatigue of the material in localized areas.

Although the plain stainless-steel liner resulted in more successful cycles of operation than did the aluminum liner, it did not approach the objective of 25 successful cycles that was arbitrarily set for the investigation. Furthermore, the random buckling that was observed for both plain metal liners was not desirable; it was believed that this type of buckling would lead to even lower cyclic life in the presence of liquid hydrogen at a temperature of -423° F. It was, therefore, believed necessary to evolve a type of metallic liner with physical or mechanical design features that would permit the liner to have elastic extensi-

bility of the order of 3 percent. Such metal liner concepts incorporating embossed patterns in the liner are discussed in the next section.

Embossed liners: Because of the apparent need for a metallic liner to incorporate some mechanism to permit the liner to extend without exceeding the elastic limit of the liner material in both a circumferential and axial direction, several types of liner configurations were evolved that would permit bidirectional extensibility through use of embossed patterns in the liner. All of these concepts incorporated the same basic principle, namely, that of periodically spacing hinges within the liner so that the desired elastic extensibility might be obtained. Figure 10 illustrates the liner hinge concept. As shown in figure 10, the structure and liner section have an original length of L and the liner segment being considered incorporates two U-shaped hinges. These hinges might be corrugations or other types of embossed ridges in the liner. Elastic extensibility is achieved by permitting the hinges to deform within their elastic limits as shown schematically in figure 10. Actually the hinged liner concept has an analogy to the cylindrical metal bellows-type structure, which is commonly used to permit some kind of limited motion between ducts and pipes containing liquids or gases. For the liner application, however, the bellows concept must be extended to permit bidirectional extensibility rather than axial extensibility.

The four types of embossed liner configurations investigated herein are shown schematically in figure 11; this figure also indicates the liner material and the type of material that was used to fill and support the embossed portion of the liner. The orientation of the embossed liners with respect to the longitudinal axis of the test vessel is also indicated. It can be seen that the single spiral and the double spiral (fig. 11(a) and (b)) provide numerous hinges for circumferential and longitudinal strain. As the strain components within the liner approach more closely a direction that is parallel to the direction of the embossed convolutions, there are strains that are exactly parallel to and within the embossed convolutions. Since the convolutions extend continuously in what amounts to a direction that is coincident with some partic-

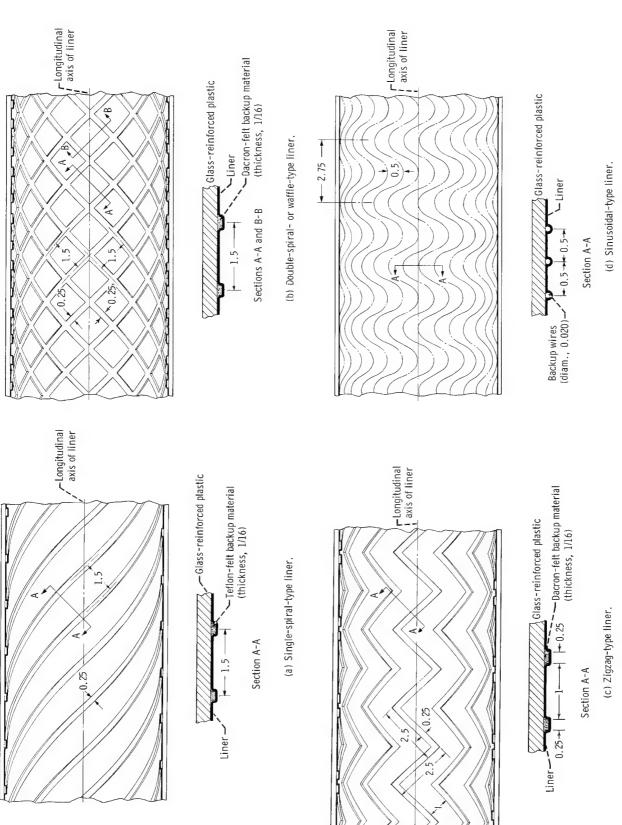
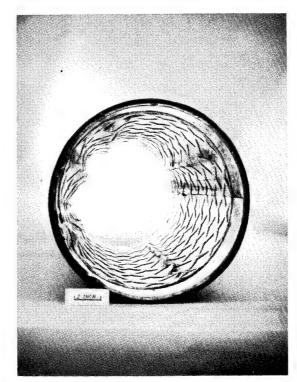


Figure 11. - Schematic diagrams of embossed liner configurations. (All dimensions in inches; not to scale,)



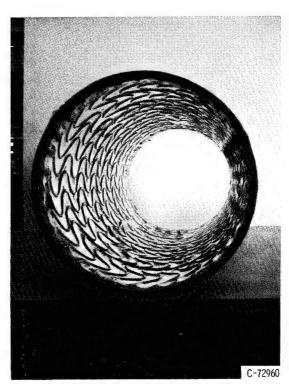
(a) Single spiral (5 cycles).



(b) Double spiral (1 cycle).



(c) Zig-zag (5 cycles).



(d) Sinusoidal (18 cycles).

Figure 12. - Embossed liner configurations after pressurization.

ular strain direction, these embossed spiral convolutions were thought to be a possible weakness and a source of potential failure for the single spiral and double spiral liner configurations. The zigzag and sinusoidal configurations (fig. ll(c) and (d)) were attempts to overcome this potential weakness in the embossed hinge concept. Both the zigzag and sinusoidal designs provide for discontinuous hinge directions with respect to any given strain direction. although the zigzag pattern does permit a strain component to be parallel and coincident with the embossed hinge, this does not occur over long uninterrupted distances. The sinusoidal configuration, because of its continuous hinge curvature and periodic reverse in curvature, essentially prevents strain components that are parallel and coincident with the hinges except for very short finite distances. In addition, the sinusoidal pattern eliminates the sharp right angle corners that exist in zigzag hinge configuration.

The results of cyclically straining and extending the various liner configurations under pressure loads that resulted in a strain of  $2\frac{1}{2}$  percent in the test cylinders is given in table II. The liner with single spiral convolutions (fig. ll(a)) was investigated first and was found to have a life of 5 cycles; although this was an improvement over that of the plain aluminum liner, it was not as good as that for the plain stainless-steel liner. A photograph of the failed spiral liner is shown in figure l2(a). A number of buckled regions at approximate right angles to the spiral convolutions are visible in addition to some randomly buckled regions. The failures in the liner appeared to originate in the ridges of the convolutions where strain components that were essentially parallel to the ridges apparently caused the aluminum foil to exceed its elastic limit. After several cycles, some of these regions where plastic flow had occurred apparently began to pinhole and then develop into a small tear causing the liner to leak excessively.

It was thought that perhaps a liner that was essentially a double spiral with the second group of spirals being at right angles to the first set might offer some improvement in cyclic life; this type of pattern results in the waffle configuration shown in figure ll(b). As shown in table II, this configuration operated successfully for only l cycle. Its mode of failure (shown in fig. l2(b)) was similar to that of the spiral convolution configuration in that the failures within the liner appeared to originate in regions where excessive plastic strain occurred in the ridges of the convolutions.

A zigzag liner configuration such as that shown in figure ll(c) was investigated next. It was intended that this design might be an improvement over the spiral and waffle patterns in that the straight sections of the ridges forming the zigzag pattern were relatively short. As seen in table II (p. 8), this configuration failed after 5 strain cycles. Figure 12(c) shows the liner at the completion of the test. The failures originated in the valleys in a manner similar to that described previously for the spiral and waffle configurations. In addition, there was evidence that pinholing was occurring at the relatively sharp right angle corners of some of the zigzags.

Based on the performance of the preceding liner configurations, there seemed to be some gain in cyclic life from spiral and zigzag types of embossed liner patterns. A liner design that incorporated some of the advantages of the

earlier designs and eliminated some of the undesirable features was then evolved. This was the sinusoidal configuration shown in figure ll(d), which was in effect a modification of the zigzag pattern.

With this sinusoidal configuration, a maximum of 18 cycles were completed before failure occurred. This liner was formed within a previous sinusoidal liner that had failed because of excessive stretching of the aluminum foil over the backup wires during pressurization. The first liner created a fillet at the juncture of the wires and the FRP tank inner wall and allowed the second liner to form without exceeding the maximum elongation capability of the aluminum foil. A cross section of this configuration is shown in figure ll(d). Figure ll(d) shows the liner at the completion of the test. Actually the failure of this liner was in the seam region and in several places where the liner was forced into a severely sharp radius at the junction of the backup wire with the wall of the reinforced plastic tank. It appears that the elimination of essentially straight and long ridges and sharp corners in the ridges improves the cyclic life of the liner.

Inasmuch as the use of the proper embossed hinge in an aluminum foil material resulted in substantial improvement of the cyclic life of aluminum liners, it might be expected that embossed hinges could also improve the cyclic life of stainless-steel liners. It was shown previously that a plain stainless-steel liner (without embossed hinges) performed satisfactorily for 8 cycles in a liquid-nitrogen environment. No attempt was made in the present investigation to evaluate embossed stainless-steel liners because of the difficulty in making suitable tooling for embossing such liners. Tooling to make embossed stainless-steel liners would have been much more costly than that for the rather crude but simple and inexpensive laboratory methods used for embossing the soft aluminum liner materials. Although no attempt was made to study the costs of fabricating any of the liners herein, it is not believed that tooling for stainless-steel liners would be so expensive that use of this material would be considered impractical.

General comments: It should be pointed out that this investigation of metallic liners has not been comprehensive and no attempt has been made herein to optimize liner design parameters. There are many variables such as spacing of the hinges, size of the hinges, shape of the hinge pattern, liner materials, and hinge backup materials that should be investigated to provide more information relative to metallic liners for application to FRP pressure vessels containing cryogens. The results reported herein provide a basis for indicating that metallic liners incorporating some type of provision for elastic extensibility of the order of  $2\frac{1}{2}$  percent have potential for cyclic operation at liquid-nitrogen temperature. The ability of these liner concepts to perform at liquid-hydrogen temperature is unknown at this time.

A factor that may have contributed to early cyclic failure of the liners investigated herein was the high liquid pressures that were required to cause the test tanks to strain to a value of  $2\frac{1}{2}$  percent. As mentioned previously in the section APPARATUS AND PROCEDURE, pressures of the order of 800 to 1000 psi were needed to obtain the desired strain; such pressures are about an order of

magnitude higher than those that would normally exist in the cryogenic propellant tanks of space vehicles. These high pressures would certainly be expected to be more severe on liner performance than pressures of the order of 50 to 100 pounds per square inch producing the same strain values. Lower pressures for a given stress level can be obtained by using cylinders with a larger diameter and a relatively thin wall. Use of larger cylinders in this investigation was precluded by the size of the cryostat available for test purposes. The high pressures used for these tests would be expected to be particularly harmful to the configurations containing the various embossed patterns because of the tendancy to deform the embossed portions of the liner considerably more than low working pressures. Even though all but one of the liners investigated herein were free to move with respect to the test cylinder wall, the very high pressures may very well have had the effect of preventing uniform slippage between the liner and the test vessel wall. If such a condition existed during the tests, it is possible that in local regions the liner extension was considerably above the average value of  $2\frac{1}{2}$  percent.

Another problem area that existed for all the liner configurations investigated were the seams that were necessary to make the liner closures. There was evidence of more pinholing and tearing in the vicinity of the liner seams than in other areas of the liner. Probably in any practical application of liners to reinforced plastic pressure vessels seams will be required in the liners. No effort was made herein to investigate thoroughly the liner seam problem, but it is apparent that more study of the seam problem is necessary.

#### SUMMARY OF RESULTS

#### Filament-Wound Glass-Reinforced Plastics

The results obtained from a preliminary investigation of the strength characteristics of some filament-wound glass-reinforced plastic materials at temperatures ranging from  $75^{\circ}$  to  $-423^{\circ}$  F were as follows:

- 1. All the materials tested showed appreciable increase in smooth tensile strength as the temperature was decreased from  $75^{\circ}$  to  $-320^{\circ}$  or  $-423^{\circ}$  F. For example, a bidirectionally wound sheet material of E-801 glass had a composite tensile strength at  $75^{\circ}$  F of 91,500 psi with increases in tensile strength of 47 and 40 percent at  $-320^{\circ}$  and  $-423^{\circ}$  F, respectively.
- 2. The material appears to be relatively insensitive to sharp notches. The ratio of sharp-notch to smooth tensile strengths of bidirectionally wound sheet material at temperatures of  $75^{\circ}$ ,  $-320^{\circ}$ , and  $-423^{\circ}$  F was 0.89, 0.95, and 0.95, respectively.
- 3. The composite tensile strength of unidirectional material of S-HTS glass wound in the form of NOL rings was about 297,000 psi at a temperature of  $75^{\circ}$  F, with increases in tensile strength of 26 and 21 percent at -320° and -423° F, respectively.
  - 4. The tensile strength of bidirectionally wound material of E-HTS glass

in the form of cylinders was 127,000 psi (based on the total composite thickness, which included circumferential and longitudinal filaments) at  $75^{\circ}$  F. The material was 61 and 56 percent stronger at temperatures of -320° and -423° F, respectively, than it was at  $75^{\circ}$  F.

5. The modulus of elasticity of E-HTS glass that was unidirectionally wound into the form of cylinders was of the order of 8,000,000 to 8,500,000 psi (based on the circumferential composite thickness) for a temperature of  $75^{\circ}$  F. An insignificant increase in the modulus of elasticity resulted when the temperature was decreased from  $75^{\circ}$  to  $-320^{\circ}$  F.

#### Liner Materials and Configurations

The results of a preliminary investigation of metallic and polymeric materials for use as liners in filament-wound glass-reinforced plastic pressure vessels indicated the following:

- 1. Aluminum foil liners having some type of provision for mechanical elastic extensibility show promise for use as permeability barriers in reinforced plastic pressure vessels subjected to cyclic pressurization (cyclic straining) with liquid nitrogen. Of the four types of aluminum foil liners investigated that incorporated mechanical means for attaining elastic extensibility, a liner having an embossed sinusoidal-type pattern was found to have the greatest durability: this type of liner was successfully cycled 18 times at liquid-nitrogen temperature to an extension of  $2\frac{1}{2}$  percent under biaxial loading conditions in a reinforced plastic pressure vessel. Aluminum foil liners incorporating embossed single spirals, double spirals, and zigzags failed after 5, 1, and 5 cycles, respectively. These results were obtained with pressure vessels that required internal pressures of the order of 800 to 1000 psi in order to obtain typical working strains of  $2\frac{1}{2}$  percent in the filament-wound glass-reinforced plastic tank wall. These pressures are about an order of magnitude higher that those that might be expected in the cryogenic propellant tanks of space vehicles to produce the same value of strain. Such high pressures probably resulted in reduced cyclic performance of the liners investigated herein because of the collapsing forces on the embossed patterns.
- 2. Plain metallic liners made of aluminum and stainless steel failed after 1 and 8 cycles, respectively, when cyclically strained to  $2\frac{1}{2}$  percent in liquid nitrogen.
- 3. Plain polymeric liners made from films of Mylar A and H-film failed to complete one full pressurization cycle at liquid-nitrogen temperature. These materials failed under biaxial loading conditions at strains that were of the order of 1 to 1.6 percent.

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Cleveland, Ohio, November 2, 1964

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